U.S. Department
of Transportation
National Highway
Traffic Safety
Administration

DOT HS 807 992
Final Report

May 1993

Evaporation Test Variability Study

TABLE OF CONTENTS

LIST OF EXHIBITS

Exhibits appear at the end of the sections where they are discussed.

LIST OF EXHIBITS CONT'D.

I. OBJECTIVES

The NHTSA conducted a study entitled, "DOT 4 Type Referee Material Brake Fluid Evaluation," Contract Number DTNH22-90-C-02067. A part of this study was to determine compliance with the FMVSS No. 116 through a round robin test by five subcontractors. A significant parameter of the FMVSS No. 116 compliance test involved an evaporation loss determination on four non-silicone type brake fluids.

In reviewing the evaporation findings, an unacceptably large variation in results was found. Analysis of the data indicated that, even with detailed instructions to the contractors, evaporation values showed a variation of 30 to 50 percent between reported results. It was concluded that the sources of this variation needed to be identified and evaporation procedure changes developed that will improve the accuracy of this test method.

The objective of the investigation work undertaken in this program was to assess the significance of pertinent parameters on the evaporation rate and devise a controlled test system that can provide acceptable, reproducible values.

II. METHODOLOGY

The existing FMVSS No. 116 procedure does not impose strict operating conditions on the wide array of variables that are capable of promoting great deviance in evaporation results. In a thermal system of this nature, it is necessary to identify the major influences that contribute to variation in results.

Evaporation is conducted in a convection oven as required in the controlling specification. Convection ovens have predictable characteristics that could, either singly or in combination, lead to inconsistent evaporation rates.

The most prominent and probable variables would involve:

- Oven configuration and design
- Temperature gradients, sensing location and regulation of the chamber heating
- Sample placement
- Chamber size in relation to surface area of the sample
- Air flow patterns.

The program was arranged so as to methodically obtain data on the variables and then to demonstrate how controlling techniques could be developed to minimize such variations. From these investigational findings, a procedure would be devised which would be capable of improving the reliability and accuracy of the evaporation rate test for brake fluids.

A round robin test schedule with three other laboratories using the modified procedure on a series of four selected brake fluids would validate the developed method and confirm its ability to obtain acceptable interlaboratory results.

The intentions of the investigational work were to utilize the existing FMVSS No. 116 procedural concepts of a heated convection oven with an exposure temperature of $100^{\circ}C \pm 2^{\circ}C$ and then to modify and control conditions of the test so as to obtain reasonably repeatable evaporation rate measurements.

To accomplish this, a series of tasks was initiated to compile data characterizing the system elements.

TASK 1 - IDENTIFY THE CHAMBER CHARACTERISTICS OF OVENS NOW BEING USED AT BRAKE FLUID TEST FACILITIES

A brief telephone survey was made, using a structured questionnaire for the ovens now in use, to obtain catalog data on make, model, size, temperature controls, location of venting ports (in and out) and placement of heating elements. This was a helpful guide to ensure that our data collection recognized the range of chambers being used. In this way, our developed procedure would be based upon "probable use" type hardware as much as the findings would permit.

TASK 2 - EVALUATE THE EFFECTS OF OVEN SIZE, TYPE OR DESIGN AND PLACEMENT OF SAMPLES WITHIN SUCH CHAMBER

Based on the oven survey information, three different oven configurations are currently used by companies conducting evaporation tests. These vary in chamber size, in air flow patterns (due to entry and venting port location) and they also have thermal gradients that could make test specimen placement critical. Thermocouple placement and oven temperature accuracy was evaluated by temperature profile measurement within the oven cavities.

Using ovens that represent the type and volume spread in general use, we determined the differences of pertinent variables between chambers so as to judge their influence on variable evaporation rates.

An oven representing each of the three designs in general use was instrumented with 27 thermocouples arrayed in three planes of nine thermocouples each. Exhibit I, attached, depicts the chamber array of these thermal detectors.

Each thermocouple was connected to a computer and readings, as a function of monitoring time, were continuously recorded. These thermal profiles were taken at the nominal 100°C temperature setting. Monitoring continued from oven start-up to the end of a 24 hour period, thus facilitating the collection of stabilized condition test data.

The data was then plotted so as to show any variations within the chambers and to help determine the adequacy of oven sensors and controls. These findings were utilized, along with other parameters from the user survey information, to obtain a good understanding of the test practice variations.

This data established how much contribution such differences exert on the evaporation rate and also defined necessary limits required to control variability.

TASK 3 - EXAMINE THE USEFULNESS AND OPTIMUM CONDITIONS OF SAMPLE ROTATION AS A NORMALIZING MEASURE TO IMPROVE EVAPORATION RESULTS

From our own limited, preliminary work and from a SAE Fluids Committee evaluation, sample rotation, using a slow turning platform within the oven, seemed to offer potential for more uniform results. A more formal data base was needed to demonstrate the merits of this modification.

Rotating shelf units were used in some typical oven styles identified in the survey and their influence was measured on the oven temperature distribution patterns and evaporation results. Rotations of 2 rpm and 6 rpm were investigated to assess the influence of rotational speed and provide a basis for selection.

TASK 4 - A METHOD OF CALIBRATING A GIVEN OVEN AND RECOMMENDED CONDITIONS FOR CONDUCTING AN EVAPORATION RATE ARE NEEDED TO ENSURE REPRODUCIBILITY

Recognizing the usefulness of standardizing an oven for product test use, a calibration fluid was sought. It would be used to adjust oven conditions to match the target evaporation rate of the calibration material and thus promote more consistent brake fluid evaporation results.

Guidelines were established for selection of such calibration fluid candidates. Many of the ovens in use have open resistance coils which could serve as an ignition point; therefore, materials with low flash points and high volatility, would not be considered. Toxicity was another criteria, as was offensive odor and corrosion. The evaporation rate should be similar to the brake fluid materials and be readily available as a material for this use.

Screening of several candidates was performed to compare their properties and test response so a useful selection could be made.

TASK 5 - USING THE BEST FIT TEST PARAMETERS FROM THE CALIBRATION FLUID DATA, DETERMINE RESPONSE FOR THE SPECIFIED FOUR BRAKE FLUIDS IN VARIOUS OVENS

Samples were run with each fluid according to the developed procedure and calibration results. This provided verification of the method for a group of actual brake fluids. The variations between runs were examined. As necessary, small tuning adjustments were made to obtain the minimum variation between replicates at both the three day and seven day test period when setting the ovens with the calibration fluid.

The four designated brake fluids used in this qualification work were:

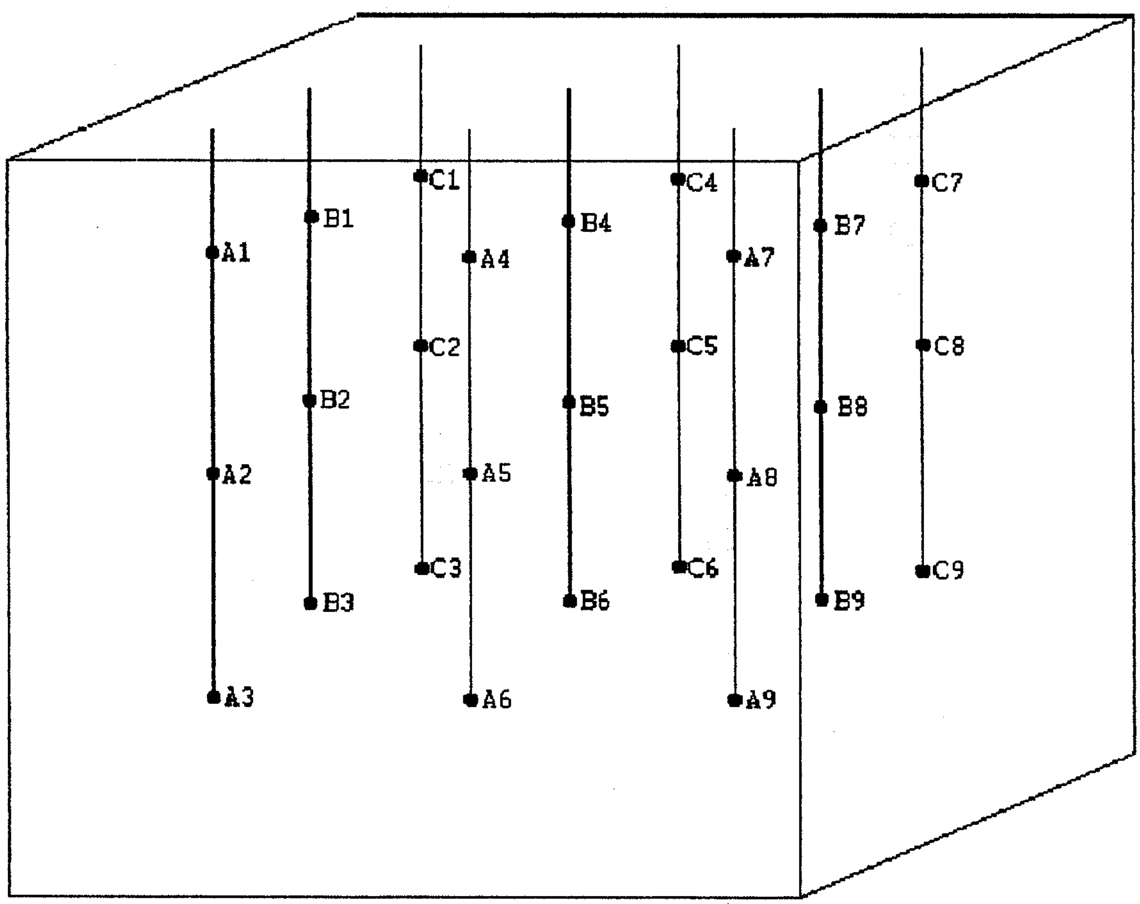
- CCL # 1 - Wagner Premium Brake Fluid
- CCL # 2 - Delco Supreme 11 Brake Fluid
- CCL # 3 - Dow 920 Brake Fluid
- CCL # 4 - Case Consulting DOT 4 Reference Fluid.

TASK 6 - CONDUCT A ROUND ROBIN EVALUATION USING THE PROPOSED EVAPORATION METHOD MODIFICATIONS WITH FOUR LABORATORIES

The designated four brake fluids would be evaluated by four laboratories, including Case Consulting Laboratories. Ovens would be standardized using the calibration fluid and oven conditions developed in the investigational work. The evaporation rates attained by each participant would be compared to establish how much improvement between interlaboratory values was achieved by the modified test procedure.

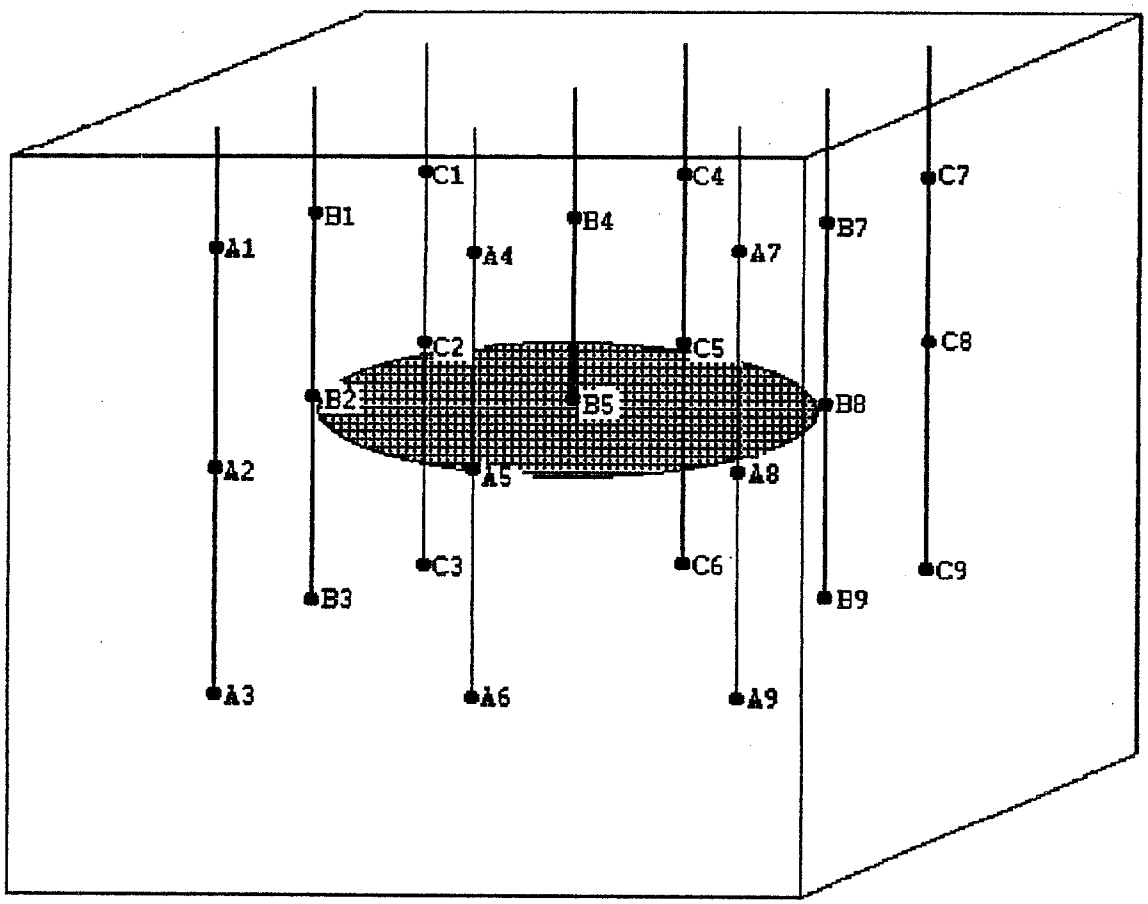
Detailed instructions and uniform data sheets were developed and supplied to each participant so as to promote uniform practices. Quantities of the calibration fluid and the other test brake fluids were shipped to them for this purpose. Calibration ranges were made available to them for the calibration fluid so they would have the target values needed to adjust their oven operating conditions.

EXHIBIT I(1)
USDOT/NHTSA
CONFIGURATION OF THERMOCOUPLE ARRAY
FOR EMPTY OVEN PROFILING



Source: Case Consulting Laboratories, Inc.

EXHIBIT I(2)
USDOT/NHTSA
CONFIGURATION OF OVEN WITH CAROUSEL



Source: Case Consulting Laboratories, Inc.

III. PROGRAM TEST RESULTS

Reported findings in this section are based upon the data developed throughout the investigational work. For convenience of handling, the task contents will be combined into four major headings:

- Oven characteristics and thermal profiling
- Calibration and procedure modifications
- Brake fluid evaluation results with modified procedure
- Round robin results.

1. OVEN CHARACTERISTICS AND THERMAL PROFILING

The oven survey provided information about the chambers in use and also their design and operating characteristics. Exhibit II, attached, summarizes the collected data.

Results of the survey revealed considerable variation in oven design, air flow patterns, location of temperature controls and points of measuring the oven temperature related to sample location. The most important influence on evaporation rate is heat and management of its functions within the test chamber.

It was necessary to understand and quantitatively measure the temperature distribution throughout the different ovens to establish:

- Uniformity of temperature
- Variations in temperature control accuracy
- Stabilization time
- Location point of chamber temperature monitor
- Optimum sample placement.

Using a pattern of three thermocouples with three planes vertically and three planes horizontal, a 27 point comprehensive temperature profile was established for each oven style. Recording continued for 24 hours to observe excursions that may occur during steady state conditions traceable to control sensitivity and response. Data was collected at 100°C as the specification target temperature.

The thermocouple network was as shown in Exhibit I. Vertical plane thermocouples were placed 2" from the bottom, midway from the bottom and 2" from the top of the oven chamber and 2" from each side, with the center plane positioned in the middle of the chamber. Horizontal planes of the thermocouples were also 2" from any oven surface.

Temperature measurements were computer controlled and recorded at short intervals of 30 seconds during oven warm-up and then lengthened to readings every 30 minutes. All the data points were computer processed to produce a summary showing the mean temperature at each thermocouple point, standard deviation and relative standard deviation for set chamber temperature of 100°C.

The profile of the three horizontal planes in each empty oven produced an understanding of variations within that oven on the bottom, middle and top horizontal plane basis and also the variation within each plane.

Exhibit III, attached, displays the plot of the average temperature of all nine thermocouples for the three horizontal planes of the three oven styles. The middle plane avoids the temperature extremes of the other two and was the logical sample location position in the oven.

<u>Stationary Shelf-Type Ovens</u>

Fisher Isotemp models were identified as being used by three laboratories for brake fluid evaporation. These have volumes of approximately 1 cubic foot for one model and 1.5 cubic feet for another, with heating elements below a bottom plate perforated by a pattern of 1/4" diameter holes. The heated air rises through these holes into the working chamber volume and out through a single slotted top vent. Two respondents monitor the temperature with a thermocouple located at 3" and 4" respectively above the sample shelf. Another participant used a thermometer at 4" above the sample shelf.

Two laboratories used the Precision Model 16 convection type oven which has an internal volume slightly more than 1 cubic foot. Thermocouples were used to monitor the interior temperature located 1" and 3" respectively above the sample shelf. This oven has a solid bottom plate with six 1/4" holes located near the front center of the plate forming a register type vent into the oven. The temperature control sensor is located in this entry heated air stream which vents through a single top slotted port.

This design promotes a different air flow pattern within the chamber than the Fisher oven which has the holes distributed across the bottom plate area.

One laboratory used a Blue M stationary shelf-type oven which had an internal volume of about 3 cubic feet. Heated air rises from an open screen bottom and had dual exit ports. Inlet air is introduced to the heating elements through grills located on the sides and rear of the oven case. A thermocouple 2.5" above the sample shelf monitored oven temperature.

Rotating Shelf Ovens

Five laboratories use a Blue M Model RS-18A-2 oven. These units have an internal volume of approximately 3 cubic feet and a rotating single shelf connected to a 6 rpm motor. Air inlet and outlets are located as in the Blue M stationary shelf oven.

Two laboratories used thermometers to measure oven temperature placed 6" above the sample shelf. Thermocouples were used by three laboratories. The locations of these placements were quite varied, reported as 0.5", 1" and 8" from the sample shelf.

Thermal Profiling of Oven Chambers

Average temperature distribution values, over a measured time period, were recorded by the arrayed thermocouples when the oven was set to produce 100°C at the thermocouple located in the center of the volume.

(1) Fisher Isotemp Ovens

Exhibit IV, attached, is a three dimensional display of the average temperatures measured at the monitored points in the oven. Compared with the center reading of 100°C point, variations as much as +3°C to -7°C were found within the oven, for a total possible temperature variation of 10°C within the same chamber.

This oven air intake is a screen located across the rear lower edge of the oven case. The average temperature values in plane "C" are cooler than the other planes, probably influenced by the inlet location. If this oven were backed up to a wall, the inlet would be restricted.

(2) Precision Ovens

Exhibit V, attached, is a three dimensional view of the average temperature recorded at the monitoring points in this oven style. Comparison of the overall point readings, with the center set point temperature of 100°C, shows a variation ranging from +5.6°C to -3.7°C, for a total oven variation of 9.3°C.

A notable item in this monitoring is the influence of the hot air flow into the oven through a small area front edge register. An average temperature in the front vertical plane as high as 105.7°C was recorded.

Air inlets for this style oven are located on the bottom as two unequal perforated rectangles. The inlet grill is only about 1/2" above the surface upon which the oven feet rest. Air flow could be easily blocked.

(3) Blue M Ovens

The stationary shelf Blue M oven, as used by one laboratory, is the same design as the Blue M oven with the rotating shelf. We removed the rotating shelf from the Blue M and used it to obtain thermal profiles of this style empty oven.

A three dimensional view of the average temperature recorded for this style oven is shown in Exhibit VI, attached. Comparing the overall point readings with the set point temperature of 100°C shows a maximum variation of +3°C and -6°C.

With the rotational unit in place, a single shelf, with its shaft attached to a rotating 6 rpm motor, is the only support surface in the oven and is located in the vertical center of the chamber.

This oven had a very open screen bottom and had dual exit ports which tended to provide a more uniform temperature distribution. Inlet air is introduced to the heating elements through three grills in the lower skirt of the oven case, one on each side and one at the rear.

A comparison of the recorded profiles for each oven shows that the Fisher Isotemp was more erratic than the Precision and Blue M units.

Tabulated average temperatures for each plane from which the plots were made are shown in Exhibit VII, attached. The standard deviations confirm the ranking of the ovens, with the Fisher standard deviation twice that of the Precision and the Blue M less than the Precision standard deviation.

From the collected thermal profiling data, the best choice sample location would be in the center horizontal plane. The plots of the empty oven thermocouple readings on this plane for each oven are shown in Exhibit VIII, attached. Using thermocouple B5, center of the middle oven plane, as the set point, the stability response of the selected set point is demonstrated in these plots.

The survey showed that the temperature monitoring practice was quite varied with respect to location. The investigational work in thermal monitoring demonstrates that temperature gradients do exist both vertically and horizontally within the oven chambers. The degree of variation differs from chamber to chamber, but the data shows that the temperature sensor placement be prescribed. The monitoring thermocouple should be placed about 1/2" above the sample dishes at B5, center of oven location. Measuring too far away from this location can result in temperature differences that could contribute to evaporation rate variations.

It was noted that, when a 6 rpm carousel turntable was installed in the ovens, some shifts in thermal profiles occurred. Air flow patterns apparently change in response to this baffle. In the case of the Blue M oven, the set point monitor registered a drop in temperature. It is part of the thermal management recommendation that the oven be adjusted to produce the set point location temperature of 100°C, with oven shelving and dishes in place before proceeding with calibration or brake fluid evaporation determinations. Comparing Exhibit VIII with Exhibit IX demonstrates these shifts.

Sample Dishes

Brake fluid samples for the evaporation tests are placed in Petri dishes which are available in two grades of glass. One grade has a uniformly flat bottom and consistent side wall height. The other lower cost grade has a bottom surface that tends to be convex which would lead to a variable thickness of fluid which can have an influence on evaporation rate.

In the interest of uniformity, the flat bottom Pyrex Petri dish will be recommended along with the other standardization items discovered in the survey and investigational work.

2. CALIBRATION AND PROCEDURE MODIFICATIONS

Calibration Fluid

From the survey response which identified the various ovens in use, the different practices in measuring chamber temperature and the thermal profiling, many items were found which would influence evaporation test results.

To bring all these variables together in some verifiable fashion would be desirable. This standardization could be helped by the use of a calibration fluid. When such a fluid is placed at a designated position in a specific oven design and temperature monitored at a defined location, there should be better brake fluid evaporation rate agreement between laboratories.

A set of criteria was developed as a basis for selecting candidate calibration fluids. To be considered, a fluid's properties should include:

- An evaporation rate similar to brake fluids
- Acceptable odor
- Low toxicity
- Safe flash point
- Readily available, reasonably consistent material.

Following a paper study of possible fluids, a few met the above requirements and were obtained for use in data collection trials. The selected fluid was a glycol ether, Dow Chemical's Dowanol TBH.

It has no offensive odor, a high flash point and an evaporation rate in the range of the brake fluid specification limit.

Evaporation determinations were made in the ovens of interest and at different oven settings to identify the response of the fluid to test conditions and to develop target evaporation values. Its evaporation rate will vary according to the temperature of exposure. In the test range, 98°C to 102°C, as currently used in the existing specification, a straight line defines the response over this range. The slope is a constant, reflecting influence of the 4°C temperature allowance within existing procedures.

Evaporation trials were made in standardized oven conditions, as determined from the thermal profiling results, to arrive at a consensus value. Exhibit X, attached, displays the calibration curve developed. Exhibit XI, attached, compares the evaporation values found when the fluid was tested in each style oven under the best condition guidelines the thermal investigation work indicated.

The data in Exhibit XI demonstrates the oven design influence on the evaporation rate of the calibration fluid under standardized operating conditions. The brake fluid test procedure and specification FMVSS No. 116 criteria for acceptance is based upon the evaporation rate after 7 days of exposure. The data shows the evaporation values have less deviation at the 7 day level.

Static oven conditions allow a ranking of oven style which shows the Fisher Isotemp oven to be the least reliable based on the calibration fluid response. Its value is much lower than the other test ovens and the variance is higher. These results reflect the more erratic temperature profile results shown earlier in Exhibit IV.

The most uniform response was found with the Blue M, 6 rpm sample rotation system. The Precision oven style was the next best, producing a 7 day evaporation rate similar to the Blue M system with less variation than the Fisher Isotemp oven, but not as close as the Blue M.

Based upon these comparisons, limited further exploration was done with the Fisher Isotemp oven. An evaluation was made to determine what improvement might be expected from the use of a 6 rpm turntable in this style oven.

Influence of Rotational Speed

Earlier preliminary experimental work demonstrated sample rotation within the oven was beneficial in reducing the variation between replicate dishes within the same sample and generally produced more consistent results with multiple sample checks.

Preliminary work used a 6 rpm external motor connected to a shaft holding a platform within the oven. It is also noted that Blue M makes a standard oven with a 6 rpm motor and rotating shelf which can be purchased as a unit.

In this program, one of the variables under consideration was the influence of speed to verify if 6 rpm was a reasonable selection. Higher speeds were ruled out as this would obviously increase the velocity of air over the fluid samples and hasten evaporation. Commercially available simple shaded pole gear-motors offered speeds of 1, 2, 4 and 6 rpm in designs suitable for oven use. A 2 rpm motor was selected for comparison with the 6 rpm.

The calibration fluid evaporation rate was determined in a Precision oven fitted with a 2 rpm carousel and with a 6 rpm carousel. Results are summarized below for 100°C set point:

	Evaporation Value, %	
Test Period	2 rpm	6 rpm
3 days	46.7	42.9
7 days	86.3	82.9

Blue M 6 rpm value were 41.4% at 3 days and 81.8% at 7 days. The 6 rpm rotation results with the Precision oven were in closer agreement to the Blue M 6 rpm values than the 2 rpm. The 6 rpm condition was selected.

Exhibit XII, attached, compares the results of the calibration fluid in the Precision static oven and the Blue M oven with 6 rpm sample rotation for 3 day and 7 day exposures at 100°C. The ovens were set for 100°C ± 1°, as monitored by a thermocouple at the oven center, about 1/2" above the sample dishes. Multiple runs were made to observe the range of variation that can occur.

Average calibration fluid evaporation values agree rather well, but examination of the precision shows the superiority of the Blue M design and sample rotation. Variance from the average for the Blue M rotation system is approximately 1/3 of that experienced in the static oven under the same temperature conditions.

Based upon this data, the calibration fluid has a tentative nominal evaporation rate of 41.4% at 3 days and 81.8% at the 7 day point based upon the 6 rpm sample rotation in the Blue M oven. The variance at 7 days is within 5% of the mean values which is quite good for a test of this type, considering all of the system variables.

Static oven values show a variance of 14% for the 7 day values and 26% for the 3 day exposure results. Variance in this use is the maximum numerical difference between the high and low evaporation values in any given test run.

The present FMVSS No. 116 Specification allows a tolerance of ± 2°C for the nominal 100°C evaporation test temperature. Limited experimental work was done in an attempt to establish the influence this temperature range would have on evaporation values. Using the calibration fluid, evaporation rates were determined at 98°C and 102°C with a static oven and the Blue M 6 rpm rotational unit as used with the 100°C data base.

Average 3 day evaporation rates of 42.5% were found for the static oven at 98°C, but with variance of about 15%, and 82.9% average for 7 days, with a variance of about 8%.

Comparative Blue M 6 rpm values at 98°C produced lower average values, 35.2%, with a variance of about 3% for 3 days and 74.3% at 7 days, with a variance of about 3%.

In similar fashion, 102°C trials showed a pattern of fairly close values between the two ovens, but a somewhat lower variance with the Blue M 6 rpm conditions than with static oven use.

The data recommends that the oven set point temperature be held at 100°C ± 1° as closely as possible to maintain reproducible results. If the calibration fluid values fall outside the established range, oven temperature may have to be adjusted to normalize conditions. In all cases, a set temperature should be held to ± 1°C.

3. BRAKE FLUID EVALUATION RESULTS WITH MODIFIED PROCEDURE

The round robin portion of the program would be the basis for ultimate judgement of the improvement in evaporation rate measurements achievable between laboratories. To augment that correlation data, on a one laboratory demonstration basis, evaporation data was developed for each of the four fluids to be used in the round robin as tabulated in Exhibit XIII, attached.

Evaporation data was acquired for the four fluids using the recommended changes at test conditions in the Precision oven and the Blue M 6 rpm chamber all at 100°C. These ovens had been standardized with the calibration fluid prior to the sample determinations.

The data shows average values to be in reasonably good agreement between the two oven styles for three of the fluids and much less acceptable agreement on the fourth fluid. Examination of the precision within each set of values clearly defines the advantage of the controlled sample rotation. Interlaboratory agreement was then established with the round robin findings.

4. ROUND ROBIN RESULTS

The following four laboratories participated in the round robin study:

- Case Consulting Laboratories, Inc.
- Dow Chemical Company
- General Motors, Delco Moraine Division
- Union Carbide Corporation.

In this program, the following brake fluids were evaluated according to the modified evaporation test procedure:

- Wagner Premium Brake Fluid
- Delco Supreme 11 Brake Fluid
- Dow 920 Brake Fluid
- Case Consulting DOT 4 Reference Fluid.

All laboratories received samples of the calibration fluid (Dowanol TBH) along with the four above brake fluids. The participants were provided with written, detailed procedures on how the test was to be conducted as well as data sheets and temperature recording logsheets. Examples are shown in the Appendix.

The modified evaporation method contained these parameters:

- A Blue M Model RS-18A-2 equipped with a 6 rpm carousel shelf was used
- The test was conducted at $100^{o}C \pm 1^{o}C$ with recommended temperature sensor location
- The oven was first calibrated by use of the calibration fluid (Dowanol TBH)
- Oven calibration was considered complete when a 7 day exposure average evaporation loss of the calibration fluid was between 78 to 86 percent
- This oven temperature setting was then used in the evaporation determinations with the 4 selected brake fluids
- Each of the 4 fluids were tested for their evaporation loss. The Petri dishes were weighed after 3 and 7 days and evaporation loss calculated.

The data from the round robin test program was computer tabulated and reviewed to establish the level of agreement that was achieved. The tabulations upon which the values were analyzed for precision and reproducibility are shown in Exhibits XIV through XVIII, attached.

Reported values demonstrate a dramatic reduction in evaporation loss differences between laboratories when this program developed procedure was used.

Some significant overview statements will highlight the improvement documented in the tabulated data:

- The highest average difference in percent evaporation loss between 4 cooperating laboratories is 5.3 on 1 brake fluid. The average difference for the remaining 3 fluids ranges from 2 to 3.8 percent between all laboratories

- Precision, as measured by a standard deviation of less than 1 percent between samples for any of the brake fluids tested, is excellent. Our studies show that in a typical static oven (Precision Model 16), the standard deviation would be 2.2 percent with a percentage loss range between replicates up to 5.3 percent

- An additional comparison in precision between the new evaporation method and the present can be further shown from another round robin study. In our report dated May 1991 entitled, "DOT 4 Type Referee Material Brake Fluid Evaluation," the CCL-4 brake fluid was tested for evaporation loss by 6 laboratories. Shown below are the results established at that time for this fluid using the old method compared with the new procedure.

-Percent Evaporation Loss, CCL-4 Brake Fluid (7 Days)-

Laboratory	Old Method	Laboratory	New Method
A	51.0	A	59.3
B	64.1	B	62.3
C	50.2	C	57.0
D	42.8	D	60.2
E	45.1		
F	57.0		

Note: Laboratories designated above using old method are not the same as those participating in the new method.

In summary, the use of the rotating shelf in an oven used for evaporation testing along with defined temperature monitoring and oven calibration fluid increases test precision results. This test system produces a significant improvement in the reliability of the results and most importantly, results can be duplicated between laboratories with a technically acceptable level of variance.

This evaluation program established that the prescribed method has been validated by the round robin tests. This validation demonstrates that higher precision and reliability are achieved following the developed procedure.

5. ADDITIONAL WORK

As part of our assignment, we investigated modifying static type ovens with a 6 rpm carousel shelf. The study showed that the two most used static oven styles were the Precision Model 16 and the Fisher Isotemp 200.

The two major components required for this modification are the 6 rpm motor and the shelf. Each item can be purchased from the following sources:

Item	Source
6 rpm Motor Model 3M104	W.W. Grainger Inc. Route 15 Richard Mine Road Wharton, New Jersey 07885 (Branches in major cities)
Shelf, Part No. M04-E102	Blue M Manufacturing 2218 West 138th Street Blue Island, Illinois 60406

A fabricated shaft is needed to attach the shelf to the motor. Additionally, a fixture will have to be provided to mount the motor on top of the outside of the oven. Shaft length is sufficient to place the shelf at the designated position in the oven cavity.

Exhibits XIX through XXII, attached, show the evaporation loss of the four brake fluids used for the round robin test in three oven styles. The ovens used were the Precision Model 16 in static condition, then modified with a 6 rpm rotating carousel shelf and a Fisher Isotemp 200 modified to include a 6 rpm rotating carousel.

In a static condition, the Fisher oven showed the most severe oven temperature profile gradients. This oven would be expected to produce the poorest precision for evaporation loss in the static condition. Collected data using the calibration fluid confirmed that this was true. The brake fluids were tested in this oven when the oven was modified with the rotating shelf and the results are shown in referenced exhibits.

All of the ovens were first calibrated using the calibration fluid before they were used to test the brake fluids.

Results demonstrate that the Precision and Fisher type convection ovens can be successfully modified to produce consistent and acceptable evaporation loss results when used in the recommended procedure. These values are in very good agreement with those obtained in the Blue M rotating shelf unit.

Such findings provide an option to the purchase of a new oven in laboratories with these convection models.

EXHIBITS II THROUGH XIII:

OVEN SURVEY SUMMARY

THERMAL PROFILING

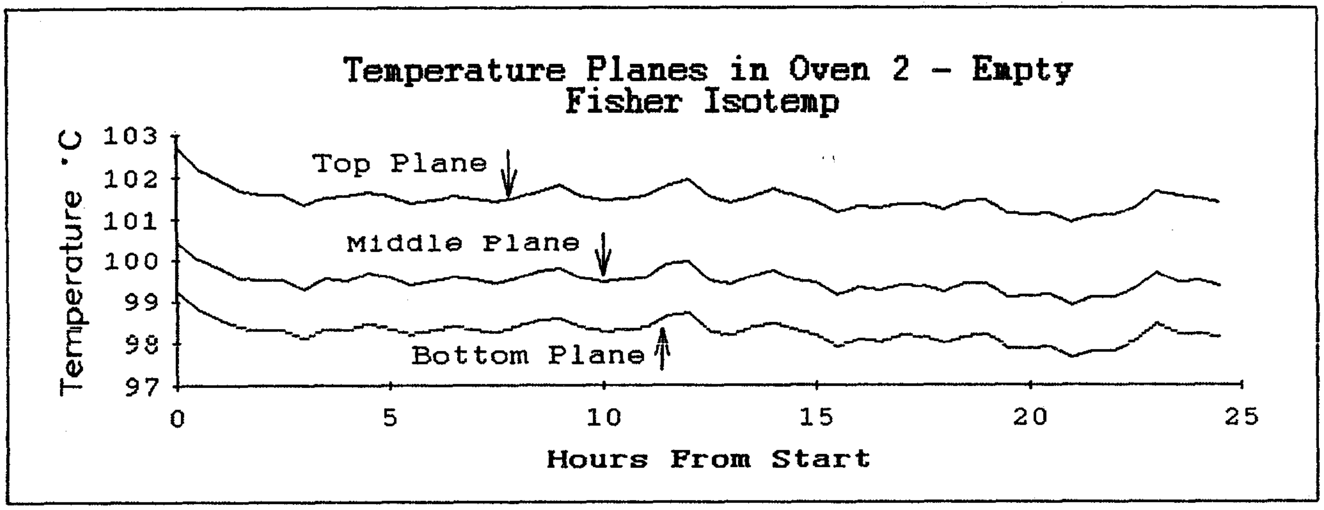
CALIBRATION AND STATIC OVEN COMPARISONS

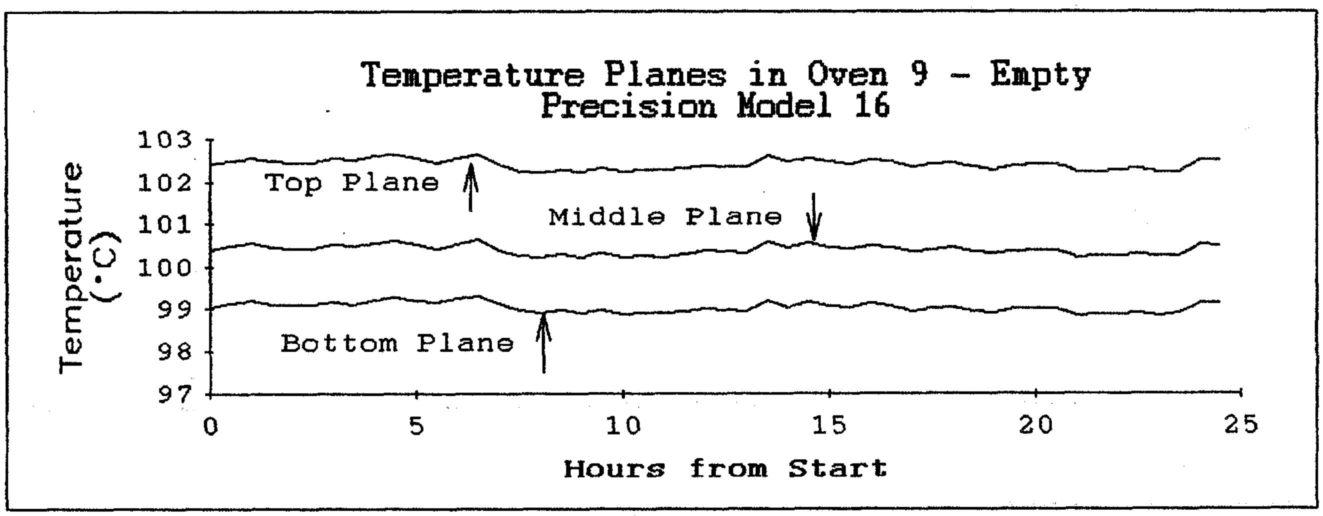
EXHIBIT II
USDOT/NHTSA
OVEN SURVEY SUMMARY

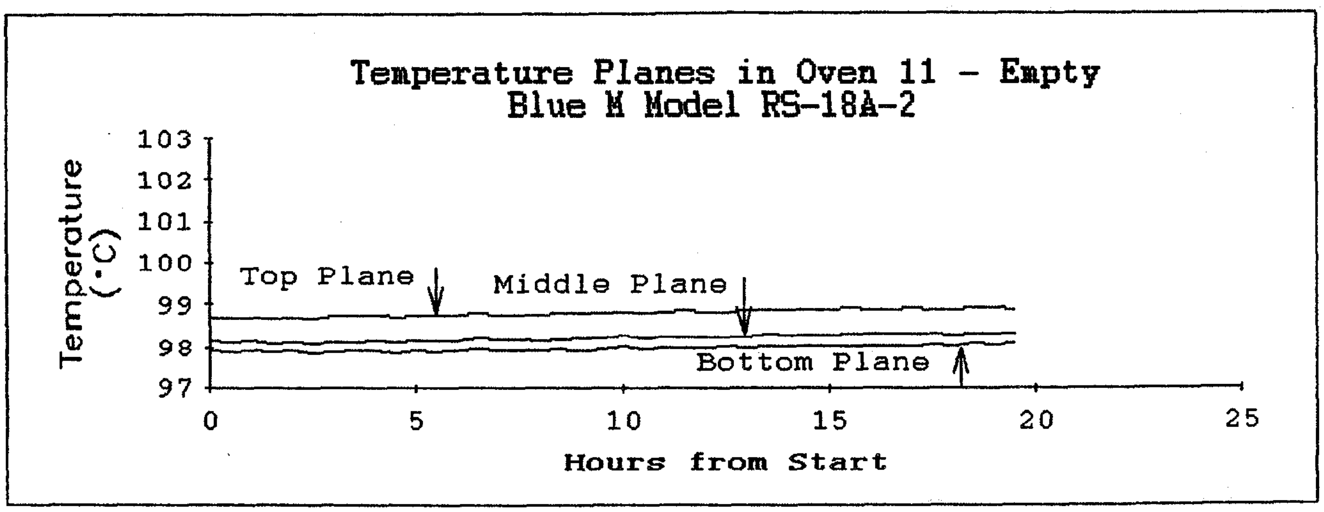
	OVEN STYLE				
	ROTATING SHELF	STATIONARY SHELF			
	Blue M Model RS-18A-2	Fisher Isotemp 200	Fisher Isotemp	Blue M Model OV-18A	Precision Model 16
Number of Labs	5	2	1	1	2
Chamber Dimensions:					
. Depth, Width, Height	18",19",15"	14",14",13.5"	12",12",12"	18",19",15"	14",13",13"
Temperature Measurements:					
. Type and Distance from Sample Shelf	Thermocouple (3) 8", 1", 0.5" Thermometer (2) 6", 6"	Thermocouple 3", 4"	Thermometer 4"	Thermocouple 2.5"	Thermocouple 3", 1"
Heating Elements:					
. Visible, Position	Yes, Bottom	Yes, Bottom	Yes, Bottom	Yes, Bottom	No, Bottom
. Type of Shielding	Open wire grid	Bottom plate random 1/4" holes	Bottom plate random 1/4" holes	Open wire grid	Solid bottom plate with 6 1/4" holes at front center
Air Exit Outlets	Dual top	Single top	Single top	Dual top	Single top
Air Vents:					
. Inlet Position	2 sides and back	Back	Underneath	2 sides	Underneath
. Outlet Position	Top (2 holes)	Top (1 hole)	Top (1 hole)	Top (2 holes)	Top (1 hole)
Testing Shelf Distance from Oven Top	10"	7", 10"	9"	10"	10"

Source: Case Consulting Laboratories, Inc.

EXHIBIT III
USDOT/NHTSA
PLOTS OF AVERAGE TEMPERATURES FOR
THREE MONITORED PLANES

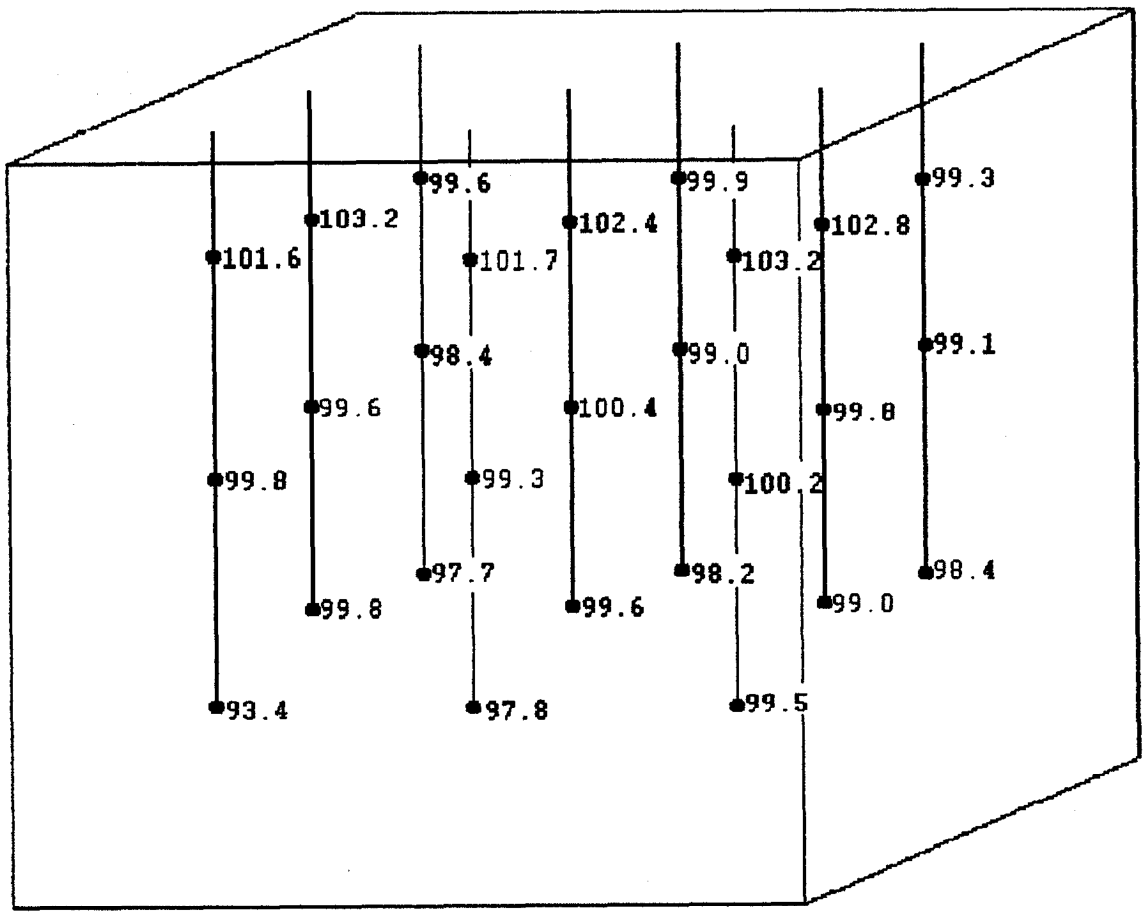






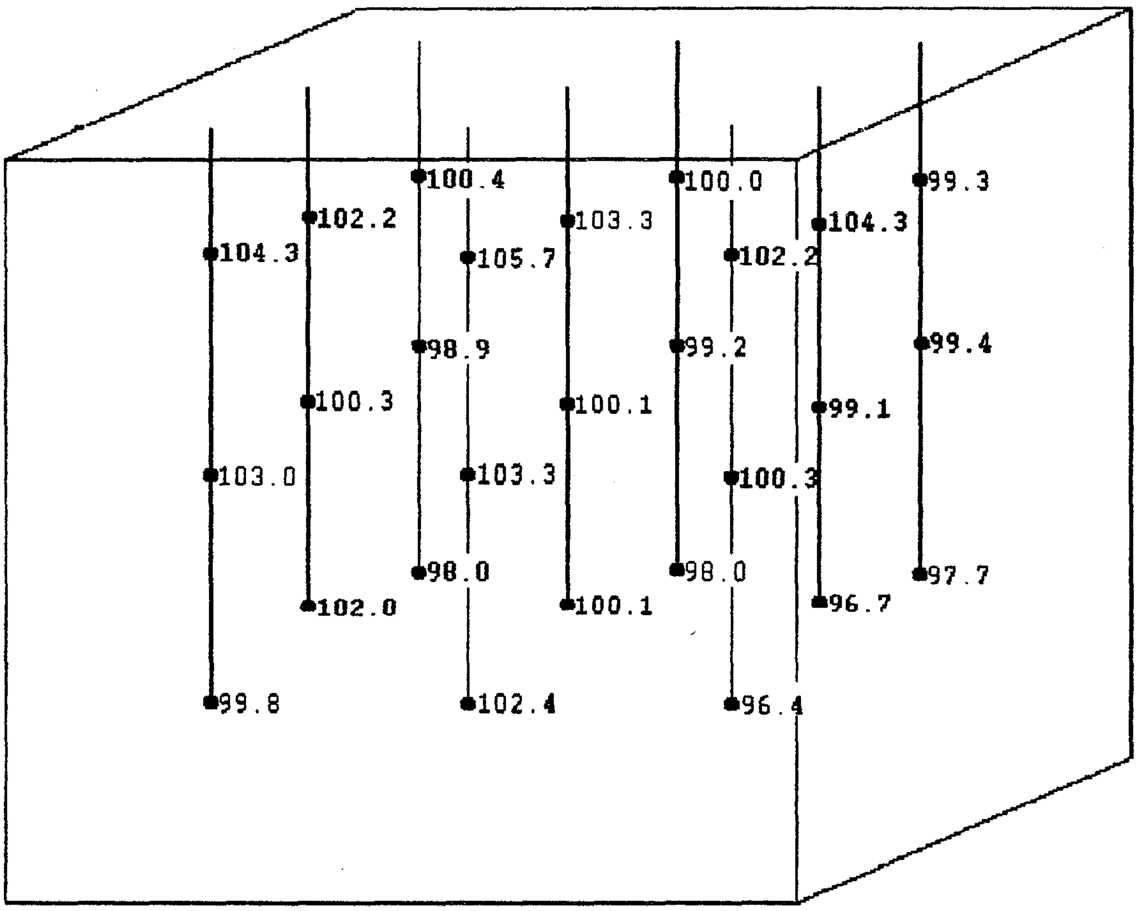
Source: Case Consulting Laboratories, Inc.

EXHIBIT IV
USDOT/NHTSA
AVERAGE TEMPERATURE AT EACH
THERMOCOUPLE LOCATION
EMPTY FISHER ISOTEMP MODEL 200 STATIC OVEN



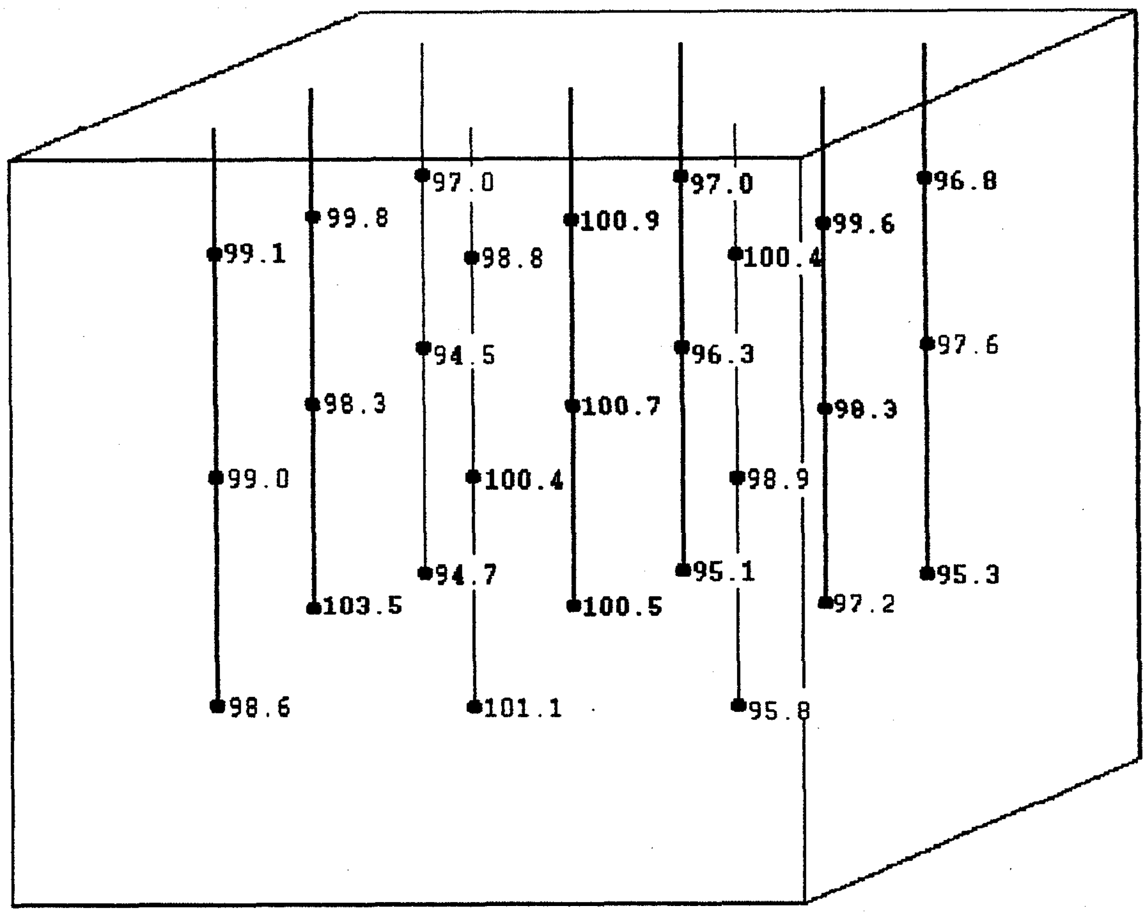
Source: Case Consulting Laboratories, Inc.

EXHIBIT V
USDOT/NHTSA
AVERAGE TEMPERATURE AT EACH
THERMOCOUPLE LOCATION
EMPTY PRECISION MODEL 16 STATIC OVEN



Source: Case Consulting Laboratories, Inc.

EXHIBIT VI
USDOT/NHTSA
AVERAGE TEMPERATURE AT EACH
THERMOCOUPLE LOCATION
EMPTY BLUE M MODEL RS-18A-2 6 RPM OVEN



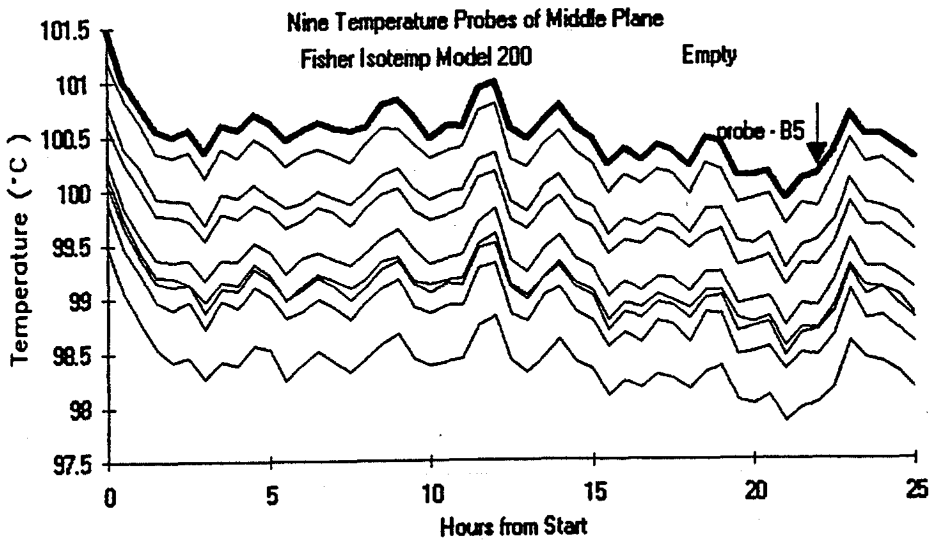
Source: Case Consulting Laboratories, Inc.

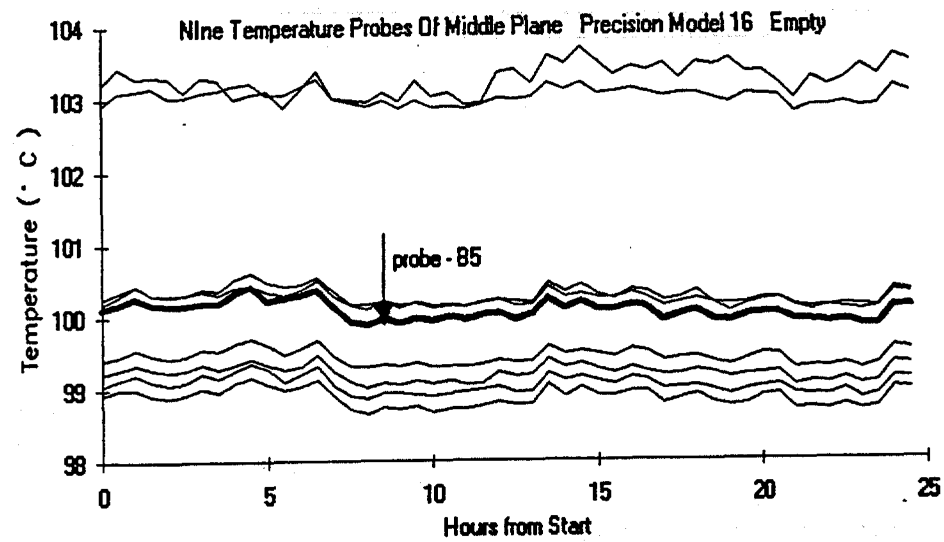
EXHIBIT VII
USDOT/NHTSA
AVERAGE OVEN TEMPERATURES FOR
EACH OVEN PLANE

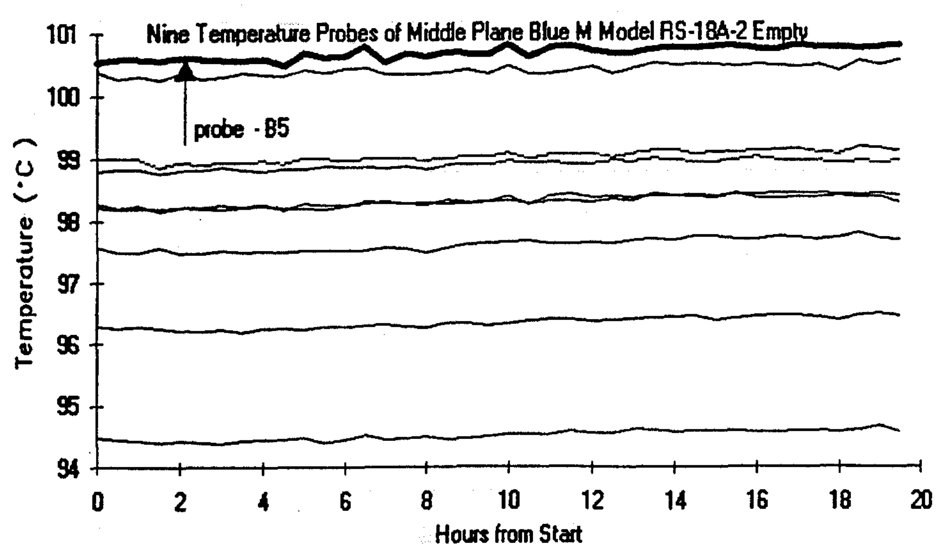
OVEN 2 Empty Fisher Isotemp				OVEN 9 Empty Precision Model 16				OVEN 11 Empty Blue M Model RS-18A-2			
Probe	Mean	SD	RSD	Probe	Mean	SD	RSD	Probe	Mean	SD	RSD
A1	101.6	0.20	0.20	A1	104.3	0.13	0.12	A1	99.1	0.11	0.11
A4	101.7	0.20	0.20	A4	105.7	0.12	0.12	A4	98.8	0.07	0.07
A7	103.2	0.21	0.20	A7	102.2	0.14	0.13	A7	100.4	0.07	0.07
B1	103.2	0.19	0.19	B1	102.2	0.13	0.12	B1	99.8	0.08	0.08
B4	102.3	0.27	0.27	B4	103.3	0.13	0.13	B4	100.9	0.09	0.08
B7	102.8	0.29	0.28	B7	104.3	0.12	0.11	B7	99.5	0.10	0.10
C1	99.6	0.23	0.23	C1	100.4	0.11	0.11	C1	97.0	0.09	0.09
C4	99.9	0.21	0.22	C4	100.0	0.11	0.11	C4	97.0	0.10	0.10
C7	99.3	0.22	0.22	C7	99.3	0.14	0.14	C7	96.8	0.09	0.10
A2	99.8	0.21	0.21	A2	103.0	0.11	0.11	A2	99.0	0.08	0.08
A5	99.3	0.21	0.21	A5	103.3	0.20	0.20	A5	100.4	0.08	0.08
A8	100.2	0.21	0.21	A8	100.3	0.14	0.14	A8	98.9	0.06	0.06
B2	99.6	0.21	0.21	B2	100.3	0.11	0.11	B2	98.3	0.07	0.07
B5	100.4	0.22	0.21	B5	100.1	0.14	0.14	B5	100.7	0.09	0.09
B8	98.8	0.22	0.22	B8	99.1	0.13	0.13	B8	98.3	0.09	0.09
C2	98.4	0.21	0.21	C2	98.9	0.13	0.13	C2	94.5	0.08	0.08
C5	99.0	0.21	0.21	C5	99.2	0.12	0.12	C5	96.3	0.08	0.09
C8	99.1	0.21	0.21	C8	99.4	0.12	0.12	C8	97.6	0.10	0.10
A3	93.4	0.24	0.26	A3	99.8	0.15	0.15	A3	98.6	0.08	0.08
A6	97.8	0.26	0.26	A6	102.4	0.16	0.16	A6	101.1	0.12	0.12
A9	99.5	0.22	0.22	A9	96.4	0.18	0.18	A9	95.8	0.06	0.06
B3	99.7	0.21	0.21	B3	102.0	0.11	0.11	B3	103.5	0.12	0.12
B6	99.6	0.21	0.21	B6	100.1	0.14	0.14	B6	100.5	0.10	0.10
B9	99.0	0.23	0.23	B9	96.7	0.15	0.16	B9	97.2	0.08	0.09
C3	97.7	0.21	0.21	C3	98.0	0.12	0.12	C3	94.7	0.07	0.08
C6	98.2	0.22	0.22	C6	98.0	0.12	0.12	C6	95.1	0.07	0.08
C9	98.5	0.21	0.21	C9	97.7	0.14	0.14	C9	95.3	0.08	0.08

Source: Case Consulting Laboratories, Inc.

EXHIBIT VIII
USDOT/NHTSA
OVEN STYLE COMPARISON OF
SET POINT TEMPERATURE STABILITY
AT 100°C

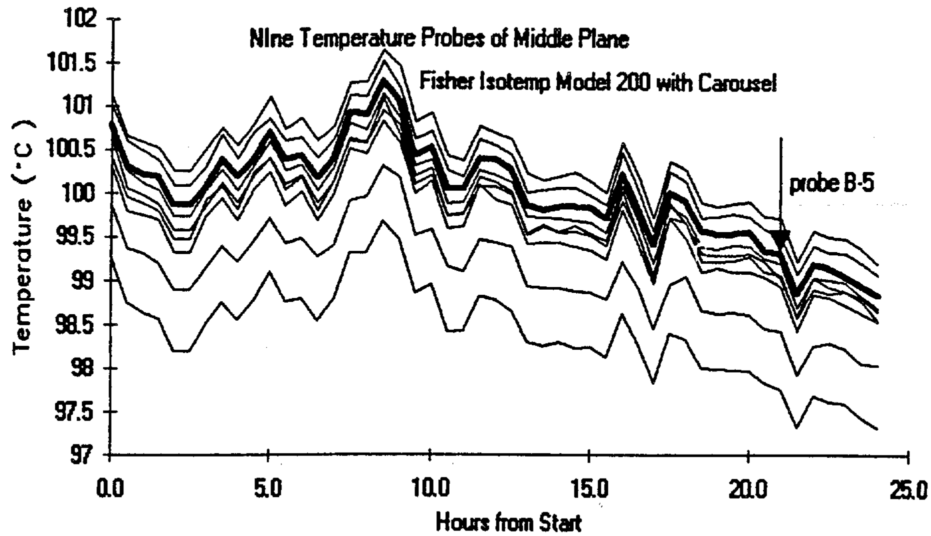


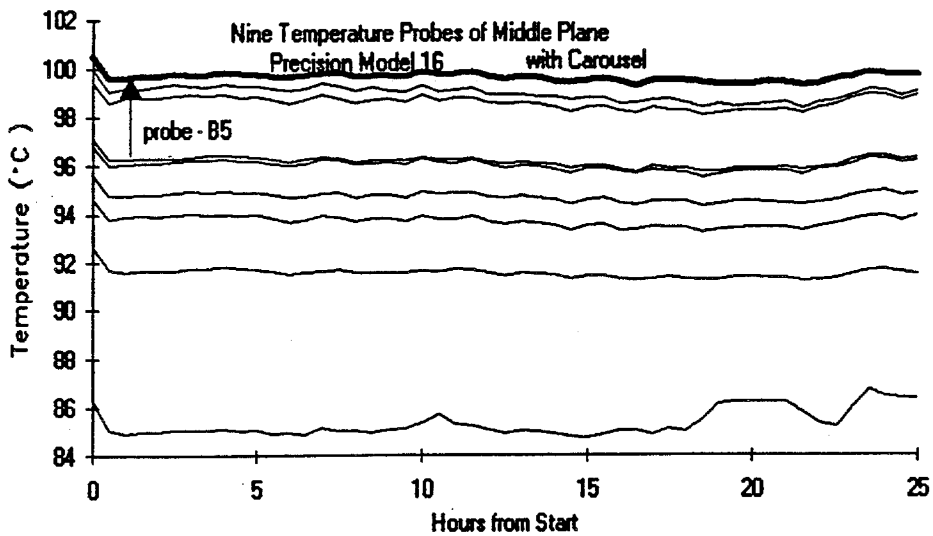


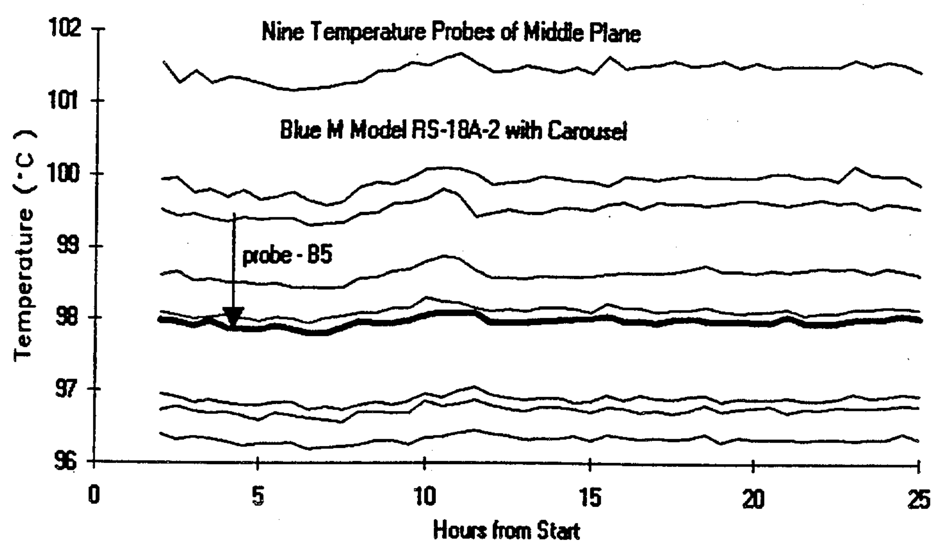


Source: Case Consulting Laboratories, Inc.

EXHIBIT IX
USDOT/NHTSA
THERMAL SHIFTS CAUSED BY CAROUSEL

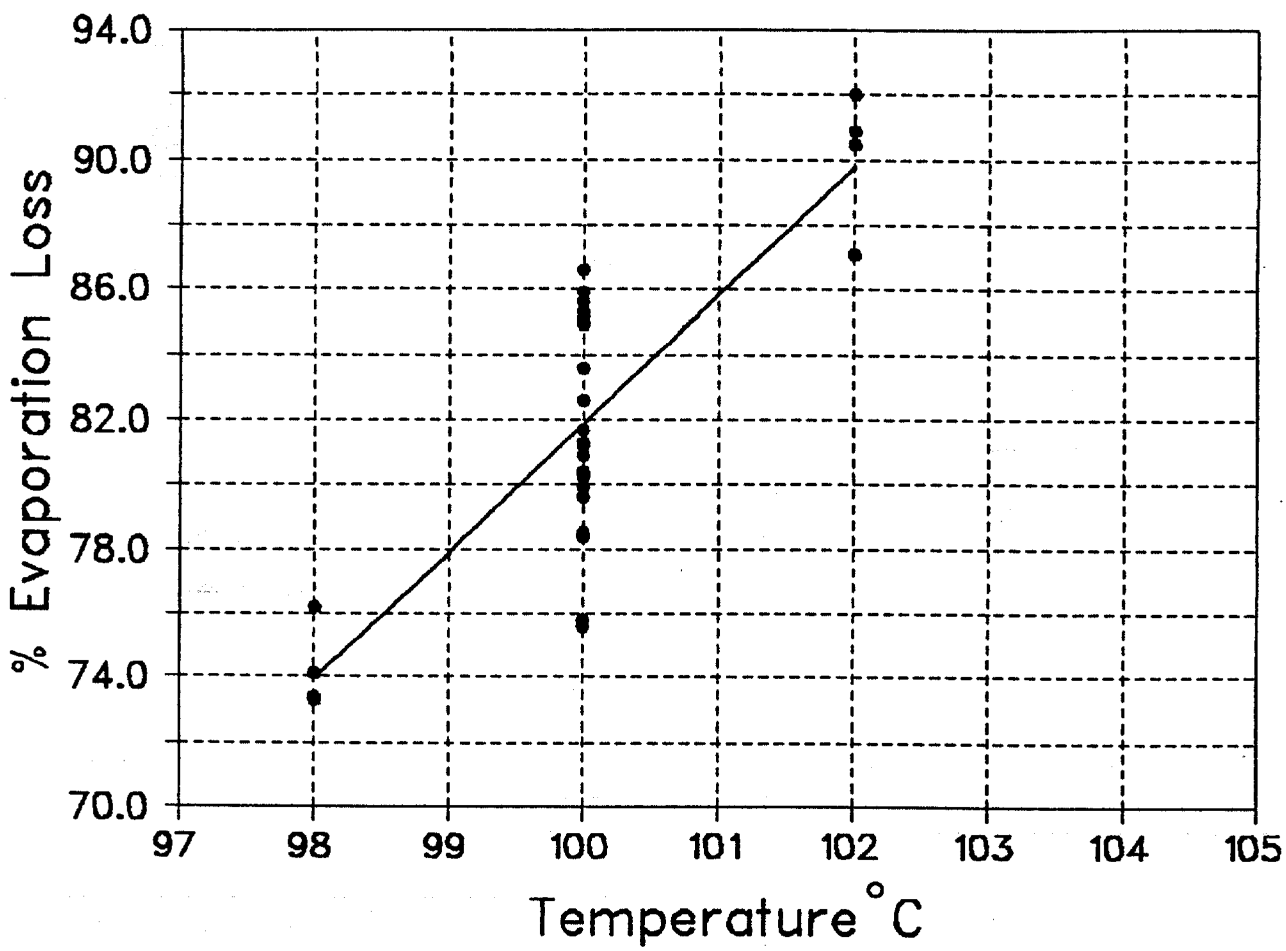






Source: Case Consulting Laboratories, Inc.

EXHIBIT X
USDOT/NHTSA
7 DAY EVAPORATION RESULTS OF DOWANOL TBH
IN BLUE M MODEL RS-18A-2 6 RPM OVEN



Source: Case Consulting Laboratories, Inc.

EXHIBIT XI
USDOT/NHTSA
COMPARISON OF CALIBRATION FLUID
EVALUATION VALUES IN EACH STYLE OVEN

Style	Temperature °C	3 Day Average % Weight Loss	High Value % Deviation	Low Value % Deviation	7 Day Average % Weight Loss	High Value % Deviation	Low Value % Deviation
Precision Model 16 Static Oven	100	39.8	30	11.1	78.4	11.2	7.2
Fisher Isotemp Model 200 Static Oven	100	28.9	15.2	15.2	62.9	15.6	12.9
Blue M Model RS-18A-2 6 rpm Oven	100	40.8	10.5	5.9	81.8	5.0	3.5

$$\% \text{ Deviation} = \frac{\text{highest or lowest value from mean}}{\text{mean}} \times 100$$

Source: Case Consulting Laboratories, Inc.

EXHIBIT XII
USDOT/NHTSA
COMPARISON OF EVAPORATION VALUES
CALIBRATION FLUID DOWANOL TBH
PRECISION MODEL 16 STATIC OVEN VERSUS
BLUE M MODEL RS-18A-2 6 RPM OVEN AT 100°C

PRECISION MODEL 16 STATIC OVEN

3 Day Values: Individual Dishes	Average	Standard Deviation	Set Range	7 Day Values: Individual Dishes	Average	Standard Deviation	Set Range
34.5 39.8 30.7 38.1	35.8	4.0	9.1	76.1 80.6 74.2 78.5	77.4	2.8	6.4
34.0 46.2 39.4 44.4	41.0	5.5	12.2	71.8 84.1 71.5 78.7	76.5	6.0	12.6
43.5 37.7 42.1 31.7	38.8	5.3	11.8	83.2 83.2 77.3 74.2	79.5	4.5	9.0
60.4 56.4 52.2 51.7	55.2	4.0	8.7	90.2 92.4 84.8 81.3	87.2	5.1	11.1
32.5 35.8 40.3 35.1	36.0	3.2	7.8	80.1 71.2 79.8 76.9	77.0	5.1	8.9
32.2 31.6 45.2 32.7	35.4	6.5	13.6	73.4 64.8 82.3 70.1	72.7	7.3	17.5
Overall Average:	40.4	4.8	10.5	Overall Average:	78.4	5.1	10.9

BLUE M MODEL RS-18A-2 6 RPM OVEN

3 Day Values: Individual Dishes	Average	Standard Deviation	Set Range	7 Day Values: Individual Dishes	Average	Standard Deviation	Set Range
39.3 39.6 38.9 39.2	39.2	0.3	.7	78.5 75.8 80.2 81.3	79.0	2.4	5.5
45.5 44.7 44.5 46.5	45.3	0.9	2.0	82.6 83.6 85.0 84.9	84.0	1.1	2.4
43.2 42.4 41.6 42.9	42.5	0.7	1.6	85.6 85.3 85.9 86.6	85.9	0.6	1.3
37.1 38.7 37.3 40.4	38.4	1.5	3.4	79.9 80.4 78.4 81.7	80.1	1.4	3.3
43.3 44.5 38.6 38.4	41.2	3.2	6.1	84.9 85.2 81.5 80.3	83.0	2.4	4.9
41.8 41.9 43.8 39.4	41.7	1.8	3.4	79.6 79.6 80.9 75.6	78.9	2.3	5.3
Overall Average:	41.4	1.4	2.9	Overall Average:	81.8	1.7	3.8

Source: Case Consulting Laboratories, Inc.

EXHIBIT XIII
USDOT/NHTSA
EVAPORATION RESULTS OF FOUR BRAKE FLUIDS

BLUE M MODEL RS-18A-2 6 RPM OVEN WITH CAROUSEL

	CCL-1 Wagner Premium		CCL-2 Delco Supreme 11		CCL-3 Dow 920		CCL-4 Case DOT 4 Reference	
	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss
	61.0	77.1	61.6	75.2	63.1	74.9	46.4	59.7
	62.5	78.3	62.7	75.7	64.0	75.5	46.6	59.9
	62.7	78.3	63.1	75.6	63.0	75.2	47.0	60.1
	62.2	77.8	61.0	75.4	64.0	75.7	48.0	61.1
Average:	62.1	77.9	62.1	75.5	63.5	75.3	47.0	60.2

PRECISION MODEL 16 STATIC OVEN

	CCL-1 Wagner Premium		CCL-2 Delco Supreme 11		CCL-3 Dow 920		CCL-4 Case DOT 4 Reference	
	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss	3 Days % Weight Loss	7 Days % Weight Loss
	52.4	72.0	59.7	75.5	52.6	68.4	44.8	56.9
	63.7	77.3	64.4	74.8	43.9	67.5	37.8	58.1
	58.5	74.1	61.1	76.6	57.4	72.2	43.9	57.3
	62.3	74.6	51.5	75.5	46.9	69.8	42.2	60.2
Average:	59.2	74.5	59.2	75.6	50.2	69.5	42.2	58.1

Source: Case Consulting Laboratories, Inc.